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TECHNICAL REPORT 21

Contract N9onr-85801
Project Designation NR031-364

REMOVAL OF Na_2O FROM A NaK SYSTEM USING A NATURAL CIRCULATION COLD TRAP

Mine Safety Appliances Company
Callery, Pa.

SECURITY INFORMATION

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REMOVAL OF Na_2O FROM A NaK SYSTEM USING A NATURAL CIRCULATION COLD TRAP

by

E. F. Batutis
S. L. Walters
J. W. Mausteller

Signed by: R. C. Werner
R. C. Werner
Project Supervisor

Approved by: C. B. Jackson
C. B. Jackson
Research Supervisor

Copy No. 12
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Mine Safety Appliances Company
Callery, Pa.

March 9, 1953

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ABSTRACT

The primary objective of this test was a determination of the ultimate oxide capacity of a natural circulation cold trap on a eutectic NaK system and the composition of the material in the trap at that point. Supplementary calculations are presented for the rates of oxide removal and iron transfer.

For this system the cold trap was no longer effective when the Na_2O in the cold trap reached 21% by wt., based on the cold trap contents only. An average oxygen removal rate was 0.0006 wt. % O_2/hr. (46 mg O_2/hr.), and the iron in the cold trap was in the ratio of Na_2O:Fe/64:1; the cold trap temperature was 400°F.

I INTRODUCTION

A. Authorization

This project was authorized by the Task Order of Contract N9onr-85801.

B. Statement of the Problem

The natural circulation cold trap experiment begun earlier[2] proved that oxygen could be removed from a flowing liquid metal stream simply by attaching a cold tank to the system by a single line. This experiment is an evaluation of the bulk density of NaK-Na_2O in such a cold trap, or the oxide capacity of a given volume of cold trap. Corollary data were taken on mass transfer of the stainless steel components to the cold trap and the rate of oxide removal from the stream.

C. Preliminary Work on the Problem

The sampling technique and analytical procedure are the same as used in previous experiments[1], and have continued to be satisfactory. The cold trap used in an earlier experiment[2] had been connected to the loop with ¼" pipe but it was found that this pipe would plug with oxide. By connecting the same trap with a shorter piece of ½" pipe two distinct advantages were gained: a) the oxide had a larger cross-section through which to diffuse and b) the temperature gradient along the pipe to the cold trap was reduced.

II PRESENTATION OF DATA

In conjunction with the proposed research of Reference 2 a cold trap with a larger connecting pipe was built and installed. See Figure 1. The connecting pipe was kept hot with nichrome heating cable and insulated in order to prevent plugging in the cold trap entrance. The trap was insulated for the first fifteen days of the test and, to lower the temperature in the cold trap, uninsulated for the rest of the test.

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1 Lee, R. E. and Walters, S. L., "Techniques of Sampling and Analyzing Hot Flowing Sodium-Potassium Alloys", MSA Technical Report IV (contract N9onr-85801, May 1, 1950).

2 Walters, S. L. and Batutis, E. F., "Cold Trap Removal of Oxygen from NaK Loops", MSA Technical Report X (contract N9onr-85801, Sept. 1, 1950).

The loop was charged with eutectic NaK (78 wt. % K) through the valve at the bottom of the cold trap. The NaK was circulated in the loop at 8 gpm at 1000°F for several days after which oxygen analyses showed 0.011 wt. % O_2 with the bottom of the cold trap at 540°F (this temperature is the saturation temperature[1] corresponding to 0.011 % O_2). An addition of oxygen as Na_2O_2, corresponding to 0.050 wt. % O_2, was made to the loop and when samples of the flowing stream showed the oxygen concentration had reached a constant value the next addition of O_2 was made. Table A, arranged chronologically, shows the results of these oxygen additions.

All the additions of oxygen were made to the NaK after cooling to 350°F or lower. The connecting pipe of the cold trap assembly was kept hot during this cooling. Immediately after each addition the NaK was heated to 1000°F within ten minutes to prevent collection of undissolved oxide throughout the loop.

<u>Cold Trap Contents</u>: The cold trap contents were analyzed by amalgamating the residue in the trap after the NaK had been drained.

The total NaK weight was found by adding the amount in the amalgam to the amount drained from the trap.

NaK	1.24 lbs.	(563g)
Na_2O	0.34 lbs.	(154g)
Fe	0.004 lbs.	(2.4g)
Cr	Trace	
Ni	None	

The weight percent of Na_2O from the above is equal to 21%. The iron is reported as the metal but may have been in some other form.

<u>Iron</u>: The total stainless steel surface area exposed to the 17 lbs. of eutectic NaK was 4285 cm^2, of which 490 cm^2 was cold trap surface.

Assuming the iron found in the cold trap came only from the walls of the cold trap because of the corrosion caused by the high oxide concentration, then the corrosion rate is 1.45 mg Fe/cm^2/month. If the iron came from all the stainless steel surface available, the calculated iron corrosion rate is 0.130 mg Fe/cm^2/month. The latter value is high compared with reported corrosion rates for these temperatures so it is believed that

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[1] Liquid Metals Handbook, 2nd Edition, pg. 114, Fig. 3.4 "Solubility of Sodium Oxide and Sodium Hydroxide in Sodium and NaK"

TABLE A

Wt.% O_2 Added	Tot.wt.% O_2 Added	Low.O_2 Conc. Reached (wt. %)	Tot. Time Betwn O_2 Additions (hrs.)	Tim.to reach Low.O_2 Conc. (hrs.)	Temp.at Bot. of Cold Trap
		0.011			540°F
0.05	0.05	0.012	162	?	510°F
0.05	0.10	0.015		<144	540°F
Cooled cold trap to 400°F					
		0.015	336		400±20°F
0.05	0.15	0.014	216	72	"
0.05	0.20	0.015	240	~120	"
0.05	0.25	0.014	336	48	"
0.05	0.30	0.013	288	168	"
0.05	0.35	0.023	120	48	"
0.05	0.40	0.027	144	< 120	"
0.05	0.45	0.027	192	< 192	"
0.05	0.50	0.039	72	48	"
0.05	0.55	0.035	96	< 96	"
0.05	0.60	0.045	96	48	"

Total oxygen removed from NaK stream = 0.566 wt. % O_2
Total oxygen found in cold trap = 0.520 wt. % O_2

The remainder of the oxygen can be accounted for by assuming that three inches of the cold ⅛" drain line contained 20 wt. % Na_2O.

most of the iron came from the walls of the cold trap.

Rate of Oxygen Removal

A rate of oxide removal calculated from Table A averages 0.0006 wt. % O_2 per hour. This value is derived by a) taking the lowest O_2 concentration at the end of a run, b) adding 0.050 wt. % O_2 to get the new starting concentration, c) subtracting the lowest O_2 concentration subsequently produced by cold trapping and d) dividing by the time.

$$O_2 \text{ removal rate} = \frac{(\text{Previous wt.\% } O_2 + \text{wt.\% } O_2 \text{ added}) - \text{new lowest wt.\% } O_2}{\text{time to reach new lowest wt.\% } O_2}$$

$$= 0.0006 \text{ wt. \% } O_2 \text{ removed/hr. (average)}$$

or for the 17 lb. system used here

$$= 46 \text{ mg } O_2\text{/hr.}$$

This type of derivation gave a series of values ranging from 0.0003 wt. % to 0.0010 wt. % removed per hour with an average as given above.

By plotting the oxygen concentration of the flowing NaK stream against the time since an oxygen addition was made, a series of curves was obtained as shown in Figure 2. When a charge of 0.05 wt. % O_2 was added to the loop the initial concentration was higher than the saturation value[1] at 1000°F (0.055 wt. % O_2). Accordingly, the curves in Figure 2 all start at 0.055 wt. %, but prior to this time the cold trap was functioning to bring the oxygen content down to this level. It will be noted in Table A that the lowest oxygen concentration reached after each oxygen addition became higher as the cold trap was being filled with oxide.

It should also be stated that most of the data in this experiment applies only to this system. More data is being collected on larger systems to establish a general criterion for cold trap performance.

Unknown Factors

The oxygen removal rate from the main stream could probably have been increased by adjusting factors such as cold trap geometry and temperatures; no attempt was made to do so during this experiment. Studies to be reported shortly will show that the temperature gradient in the cold trap does affect the rate of oxide removal. It is also true that the oxide concentration in the NaK stream could have been reduced to values as low as 0.003 wt. % if the cold trap temperature had been maintained at room temperatures. Although there was no

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[1] See reference 1, page 2

direct evidence to indicate plugging of the connecting pipe the geometry of the connecting pipe was such that it would have been possible to have the oxide precipitation occur there, causing partial plugging and thus affect the rate of diffusion. If this were the case the plug could have been removed eventually to the colder section of the cold trap but the overall O_2 removal rate meanwhile would have been decreased.

III PROPOSED RESEARCH

The study completed in the NaK system leads to the following questions.

1. What is the limiting bulk density in a cold trap on a sodium system and is it temperature dependent?
2. Does the rate of oxide removal from sodium differ from that of NaK?
3. Can the rate of oxide removal be increased?
4. Is there mass transfer of stainless steel components to a natural circulation cold trap?
5. Will a diffusion cold trap's effectiveness be affected by vibration or gravity; i.e., whether the cold trap is pointed upward or downward?
6. Should the cold trap be placed on a hot or cold leg?
7. What is the optimum design for a diffusion cold trap?

These problems and others are being studied. Questions such as those above will be considered in the next report upon the completion of experiments with a sodium filled loop.

IV SUMMARY AND CONCLUSIONS

As reported earlier, initial oxygen additions to a loop did not always appear in subsequent analyses and statements were made suggesting "gettering" by components in the system. Consideration of the oxygen balance in this report shows oxygen disappearance is explainable by cold trapping. In any liquid metal system, there may be some slight amount of gettering but the bulk of the oxide can be cold-trapped by drain and access lines where the sodium or NaK is at a temperature below that

of the main stream; these cold zones will act as efficient cold traps. Theoretically, cold traps could be used to remove oxide from liquid metal streams to low values (0.003 wt. % O_2) if the proper conditions were maintained. Further experiments to prove this and other points have been planned and are under way.

Analagous work using sodium has been completed and is the subject of a report to be issued shortly. The performance of natural circulation cold traps with sodium and NaK, using identical loop configurations is compared in the forthcoming report.

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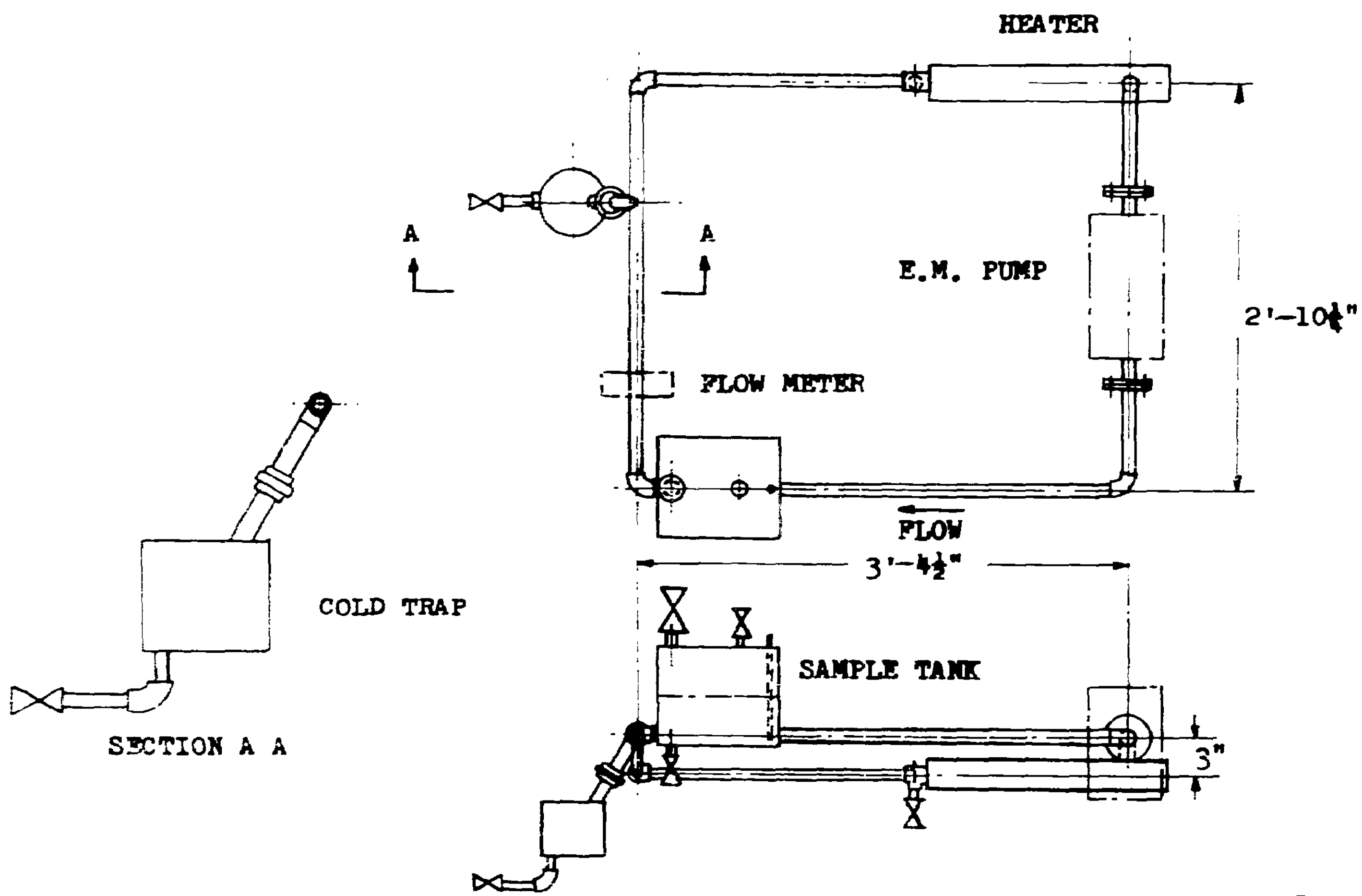

FIGURE I

REVISIONS

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